

# Development of a Super Fast-cure and Flame-retardant EVA-based Encapsulant

R.T. Tucker, S.C. Agro, and J.P. Galica  
Specialized Technology Resources, Inc.  
10 Water St., Enfield, CT 06082 USA

## ABSTRACT

One of the objectives of STR's current DOE/NREL subcontract is to optimize the extrusion process for and material properties of a super fast-cure and flame-retardant EVA-based encapsulant. It was found that two derivatives of the super fast-cure formulation are stable at extrusion temperatures. The curative package in the flame-retardant material was redesigned for more robust curing. A non-halogenated derivative of the flame-retardant formulation had comparable flame-retardant activity to the original, halogenated version.

## 1. Introduction

Under the BP Solarex subcontract ZAX-8-17647-05 awarded by DOE/NREL, Specialized Technology Resources Inc. (STR) developed two (2) EVA-based (ethylene vinyl acetate) encapsulants – a faster-curing (super fast-cure) and flame-retardant (FR) formulation. Photovoltaic modules manufactured with the super fast-cure formulation could be laminated using a six (6) minute process (105 sec. pump-down, balance press) [1, 2]. The flame-retardant formulation allowed modules to meet the Class B flammability rating under UL 1703 [1].

Although the encapsulant materials met specific performance requirements set forth by the subcontractor, there were still observable and pertinent shortcomings with the developed materials. For the super fast-cure formulation, the majority of the shortcomings were observed during the extrusion process.

- Screw slippage: the super fast-cure material did not provide adequate friction within the extruder barrel to allow the screw to properly convey and mix the material through the process.
- Scorching: evidence of crosslinking during the extrusion process was observed. Premature crosslinking resulted in uneven encapsulant film thickness and material deposits within the film and extruder.
- Compounding: an overnight procedure was necessary to mix the various additives of the formulation with the EVA resin.

In addition to processing challenges with the super fast-cure formulation, modules laminated with the encapsulant did not pass the wet hi-potential (hi-pot) evaluation of the IEC-61215 qualification test.

For the FR EVA formulation, an unsatisfactory level of crosslinking was observed, most likely due to the presence of the flame-retardant additives. The low crosslinking level prevented further testing and characterization of the material. Furthermore, the formulation contained a halogen-based additive;

halogenated chemicals are considered environmental risks, particularly in Europe. Therefore, a non-halogenated FR formulation may prove to be more acceptable and practical.

As part of STR's current DOE/NREL subcontract, one of the objectives is to address the processing and performance shortcomings of these EVA-based formulations. The purpose of this paper is to summarize the progress on the above objective.

## 2. Experimental

### *Approach*

Based on the nature and make-up of the curative package in the super fast-cure formulation, it appears that one of the curative's constituents was responsible for the observed screw slippage. It was surmised further that this same constituent was part of the mechanism that reduced the encapsulant's ability to prevent current leakage during the wet hi-pot evaluation. Therefore, reducing and/or minimizing this specific curative additive was the goal, while maintaining the fast and efficient curing properties of the overall curative package.

For the FR EVA encapsulant, a new curing system would be developed to improve crosslinking efficiency. The new system would have similar or better curing performance compared to the STR fast-cure formulation 15295P.

### *Moving Die Rheometry (MDR)*

MDR, a standard test method that analyzes crosslinking efficiency in rubber and plastic materials, was used to measure curing kinetics to access the impact of modifications to curing packages or evaluate new curing systems. The rheometer measures the shear modulus (i.e. torque) as a function of time at a set temperature during the crosslinking of a polymer. Torque development is a function of crosslink density; as the polymer crosslinks, the resistance to shear (measured as torque) increases. All MDR measurements were carried out in accordance with ASTM D 6204-97.

### *Mooney Scorch Analysis*

The Mooney viscometer is an instrument that compares the processability and stability characteristics of polymer materials. The viscosity is measured as a function of time at constant temperature. Like MDR, the viscosity is related to the curing activity of the polymer. A material that has a strong tendency to crosslink prematurely (i.e. scorch) during processing will have a higher Mooney viscosity per unit of time compared to a material that is not subject to scorching. Mooney Scorch analysis was performed at 104°C.

### *Mixing Bowl*

The mixing bowl measures crosslinking level at constant temperature like the Mooney viscometer, but a shear component is introduced that simulates more accurately the conditions within an extruder. Evaluations with the mixing bowl were accomplished using a Brabender Plasticorder/Rheometer fitted with an oil-heated mixing

bowl and high-shear blade mixers. The bowl was maintained at 104°C, and formulations were sheared at 60 rpm.

Flame Impingement

To evaluate the flame-retarding and flammability performance of candidate FR EVA formulations, a test method was modeled after 16CFR Part 1610. Experimental materials were exposed directly to a butane flame at 1,895°C for 15 minutes. During flame impingement, the material was observed for burning, charring, and/or dripping characteristics, as well as the ability of the flame to penetrate through the sample material.

3. Results and Discussion

Super Fast-cure Formulation

As mentioned above, the curing system of the super fast-cure (SFC) encapsulant was the focus of the study. To address the issues with this formulation, the overall liquid content (LC) in the curative package was systematically reduced and the curing kinetics measured. Figures 1 and 2 show MDR curves for various SFC formulations as a function of LC at 150°C, 155°C, and 160°C. The maximum torque achieved ( $S'_{Max}$ ), which is directly proportional to the crosslinking efficiency, decreased with the reduction of LC.

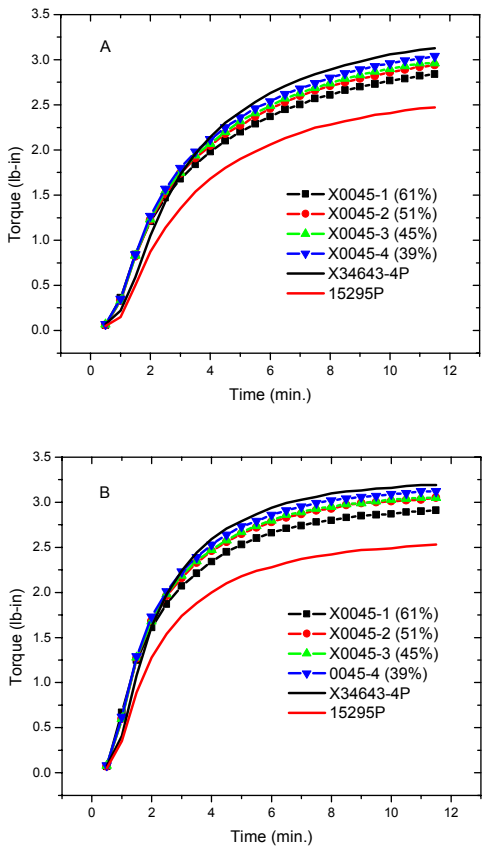


Figure 1. MDR curves for SFC formulations and 15295P at A) 150°C and B) 155°C. LC indicated in parenthesis.

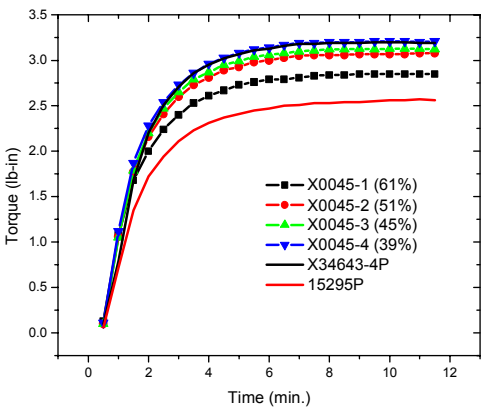


Figure 2. MDR curves for SFC formulations and 15295P at 160°C. LC indicated in parenthesis.

Table 1 compares the peak curing rates of the various SFC formulations to the original SFC encapsulant, X34643-4P, and 15295P. The SFC reformulations had slightly greater curing rates than the original SFC material. Out of the four SFC reformulations, X0045-4 had the most similar curing kinetics to X34643-4P.

Table 1. Peak curing rates (S'/min.) as a function of temperature for SFC formulations.

	X34643P	15295P	X0045-1
150°C	0.99	0.8	1.03
155°C	1.43	1.22	1.55
160°C	2.09	1.77	2.25

	X0045-2	X0045-3	X0045-4
150°C	1.06	1.06	1.06
155°C	1.61	1.56	1.59
160°C	2.38	2.37	2.52

X0045-4, X34643-4P, and 15295P were evaluated with the Mooney viscometer in order to compare the processability of each material. Figure 3 shows the Mooney Scorch data for all three formulations at 104°C. The data surprisingly showed that both SFC formulations had similar process stability compared to the control 15295P formulation, although scorching was noted to occur with the original SFC formulation during its extrusion trials.

To follow-up the Mooney Scorch analysis, a mixing bowl experiment was performed on 15295P and both SFC materials. Because process stability was the focus of this experiment, the critical measurement from the mixing bowl study was the time it takes for the material to initiate crosslinking.

For 15295P, X34643-4P, and X0045-4, crosslink initiation times were 12.5, 7.5, and 8.5 minutes, respectively. Including the shear component demonstrated a more pronounced difference between the formulations. As expected, the reformulated SFC formulation was slightly more stable (i.e. longer time to initiate crosslinking) compared to the original SFC formulation.

The maximum material dwell time in an encapsulant production extruder was determined to be five (5) minutes. Both SFC formulations had characteristic times (crosslink initiation times) greater than the residence time, suggesting that the SFC formulations should not crosslink during extrusion. Therefore, it appears that material scorching is dependent more on the flow dynamics and operating conditions of the extruder rather than the material formulation.

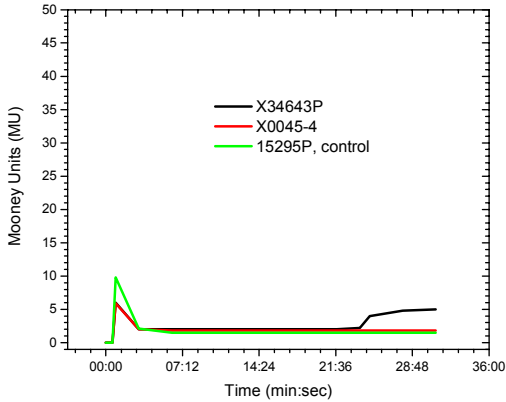


Figure 3. Mooney Scorch data for SFC formulations and 15295P.

Flame-retardant Formulation

MDR data in Table 2 and Fig. 4 compares 15295P and the original FR formulation (X33579P-FR) to two (2) experimental FR formulations (X122-11-1P and X122-11-2P). The experimental FR formulations had more robust curing performance (faster cure speed and higher  $S'_{Max}$ ) versus the other controls. In particular, the curative package in X122-11-2P greatly improved the crosslinking efficiency, resulting in higher  $S'_{Max}$  compared to 15295P and the SFC materials. Although the curing kinetics and efficiencies were improved in both FR encapsulants, these materials appeared to have adequate processing stability under Mooney Scorch (Fig. 5).

Table 2. Peak curing rates ( $S'/min.$ ) as a function of temperature for FR EVA formulations.

	15295P	X33579P -FR	X122-11- 1P-FR	X122-11- 2P-FR
150°C	0.8	0.53	0.65	1.11
155°C	1.22	1.39	0.85	1.34
160°C	1.77	1.37	1.50	2.80

A non-halogenated FR EVA formulation was evaluated for its flame-retardant effectiveness against the original, halogenated formulation. Each formulation was laminated to 15295P, and then cured prior to flammability testing.

When the flame impinged first on the 15295P side, it took less than one (1) minute for the flame to burn through the non-FR layer. The flame and subsequent

combustion appeared to increase in intensity 30 seconds into the start of the test, likely due to cleavage of the highly flammable acetate groups.

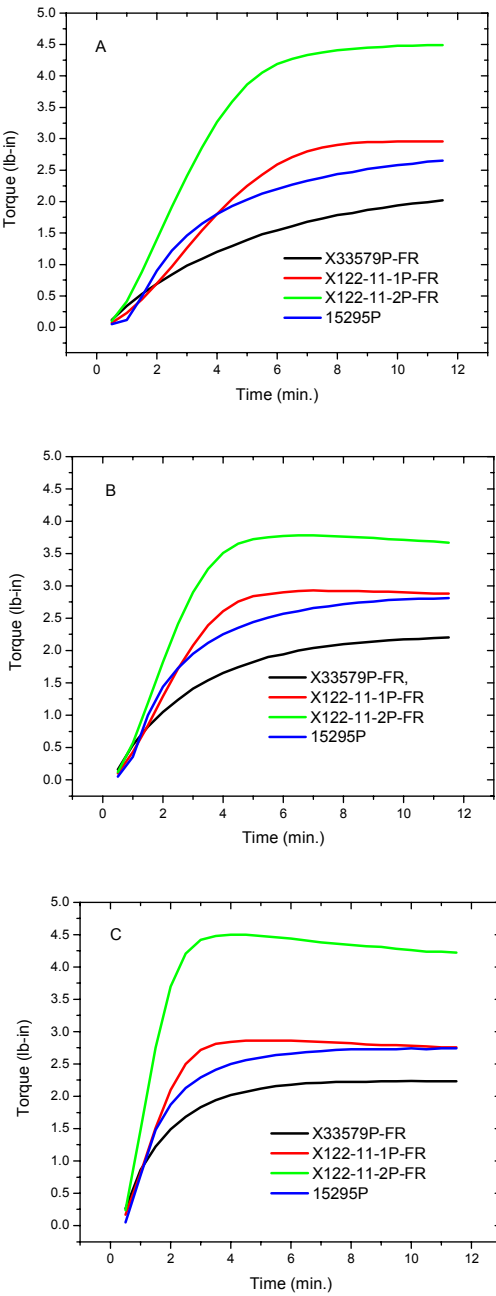


Figure 4. MDR curves for FR EVA formulations and 15295P at A) 150°C, B) 155°C, and C) 160°C.

Once 15295P was effectively burned off, the flame impingement contacted the FR EVA layer. However, for both the halogenated and non-halogenated formulations, the penetration of the flame appeared to stop at this layer. During the rest of the 15-minute exposure period, neither FR formulations burned, nor fueled the butane flame as the 15295P did. When FR EVA was exposed first to the flame (i.e. impingement first started at the FR EVA layer), the FR layer did not burn, and because the flame could not

penetrate this layer, the regular EVA layer underneath only darkened but did not ignite.

Depending on the laminate layer exposed to the flame first, the burning patterns on the laminate were comparable for both formulations. The similarity in how the material managed the flame impingement illustrated the flame-retardant effectiveness of the non-halogenated formulation.

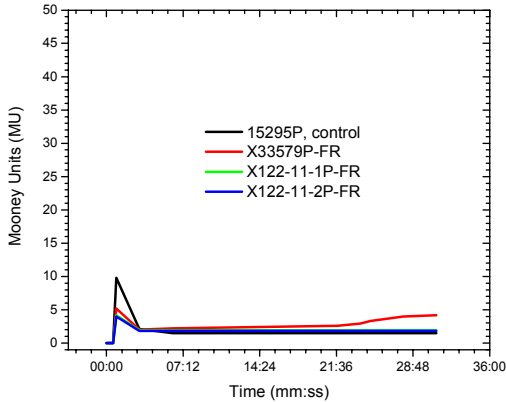


Figure 5. Mooney Scorch data for FR EVA formulations and 15295P.

4. Conclusions

In order to address the shortcomings of the super fast-cure formulation and flame-retardant EVA-based encapsulants, reformulation efforts were initiated under STR's DOE/NREL subcontract. The results to date

show that both super fast-cure formulations are stable at extrusion temperatures. Previous observations of material scorching may be more of a function of extruder setup and flow dynamics. The next step will be to identify potential areas within the extrusion process that could contribute to scorching (e.g. areas where material hold-up or flow stagnation is possible). Once identified and the process optimized, the super fast-cure candidate formulations will be manufactured, laminated into modules, and the modules evaluated according IEC-61215.

The results of the flame-retardant development efforts demonstrate that the new curative packages can significantly increase the crosslinking efficiency of the encapsulant. In addition to the improved curing system, the non-halogenated flame-retardant formulation appears to have as much effectiveness as the original, halogenated formulation. The halogen-free FR formulation with the redesigned curative package will be manufactured and evaluated in modules for flammability resistance rating.

REFERENCES

[1] Galica, J.P.; Sherman, N.; "Results to Date – Development of New EVA-based Encapsulants; Faster-curing and Flame-retardant Types," *Proceedings of 28<sup>th</sup> IEEE PV Specialists Conference, 2000, Anchorage, AK.*

[2] Galica, J.P.; Sherman, N.; "Development of Low Cost Faster-curing Encapsulants for Terrestrial PV Modules," *Proceedings of 16<sup>th</sup> European PV Solar Energy Conference, 1999, Glasgow, Scotland.*